INDUSTRIAL HYGIENE SAMPLING GUIDE
FOR CONSOLIDATED INDUSTRIAL
HYGIENE LABORATORIES
(CIHLs)

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## SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

(See Biological Samples Section for requirements on shipping.)

1. Small sample media such as sorbent tubes and filter cassettes should be bound together (i.e., rubber band) or placed in plastic bags to reduce the possibility of being overlooked or discarded. Sample cassettes and sorbent tubes should NOT be wrapped in tape. Simply affix a legible sample submission number (preferably a preprinted label) to the sample and neatly package it to avoid shipping damage. Never ship air samples and bulk samples in the same shipping package.

2. Submit separate request forms for each type of analyses as follows: Segregate and ship your samples in individual categories of air, bulk, wipe, and biological samples subdivided by metals and organics.

3. Request for analytes should not exceed 3 or 4 organic compounds per sorbent tube. Always check compatibility information on page 13 to ascertain the contaminants collected are compatible with each other and with the analytical procedure. Call the laboratory regarding compatibility when in doubt. Filter samples submitted for metal analyses by atomic absorption spectrophotometry to all CIHL should not contain more than 3 or 4 metals per sample. Since all CIHLs are constantly upgrading analytical instrumentation, check with your lab before submitting a sample for up to ten metals using ICP. Please list the requested metals in order of priority.

4. When requested, ship small quantities of bulk organic solvents in screw cap (Teflon lined) glass vials (e.g., 7.4 milliliter vials, Supelco # 2-3295; or 15 milliliter vials, Supelco #2-3296) to assist the chemist in the analysis of the air samples. Prior to shipment place a permanent ink mark at the level to which the vial is filled. This allows the chemist to determine potential leakage during shipment. Rarely will more than 5.0 milliliters be required. Never ship the bulk and air samples in the same shipping package. Provide information telling the chemist which bulk sample corresponds to the air samples.

5. Most determinations require a minimum of two blanks or one blank for every ten samples submitted, whichever is larger. Remember to always provide 20-30 ml of unexposed impinging solution to be used by the laboratory as reagent blanks and in quality control. The blanks are analyzed by the CIHLs and reported as micrograms (ug) "contaminant" per sample (e.g., per filter, per tube, etc.).

6. All references to water in this guide are deionized or double distilled water.

7. When submitting elemental analysis of hard metal alloys, the bulk sample must be in the form of fine filings, powder or very thin wire.

8. The preferred refrigerant for samples that require refrigeration is freezer packs or frozen gel blocks. Ice may be used for hand-carried lab samples, however the ice must be doubly wrapped in plastic zip-lok bags to avoid leakage. Never use ice when shipping by U.S. Postal Services or commercial delivery services.

9. Shipping containers should be appropriately labeled such as "Fragile", "Refrigerated Material", "Liquid Samples", "Etiologic Agent/Biomedical Material", etc.

10. All samples and materials being packaged, labeled and shipped are governed by Federal, State and local regulations. Compliance with these regulations is the responsibility of the person submitting the samples.

11. In the case of unusually large shipments or high priority samples, please contact the laboratory prior to submission (i.e., as a "heads-up").

## SAMPLE TURNAROUND TIMES

Samples will be analyzed on a "first come, first served" basis. Urgent samples will be given special priority and analyzed in one to three working days when the laboratory has been notified in advance of the shipment and when the samples have arrived by special shipment or priority mail. Most routine samples will be analyzed within 10 working days after receipt of the sample. If you have not received your analytical report after 20 working days, please notify the laboratory and check on the status of the samples.

## SAMPLE COMPATIBILITY

Since sampling and analytical methods are normally evaluated for a single analyte, care should be given in the interpretation of the methods' CV. When in doubt concerning multi-components samples, take individual samples. The following compounds require special processing for analysis and consequently cannot be analyzed for other compounds in the same sample:

Acetic Acid
Acetonitrile
Acrolein
All cellosolves can be analyzed on same tube, e.g., butyl-, methyl-, etc., however these cellosolves and common organics are not compatible.
All impinger analytes
All isocyanates
Ammonia
Aniline
2-Butanone
Butyl Cellosolve
Camphor
Cellosolve
Chlordane
Chromic Acid or Chromium (VI)
Coal Tar Pitch Volatiles
Cresols
Ethylene glycol

Ethylene oxide
Ethyl ether
Formaldehyde
Hydrazine
Methanol
Methyl Cellosolve
Methyl methacrylate
2-Nitropropane
PCBs
PGDN (Otto Fuel)
Phenol
Pyridine
Tungsten

The following groups of compounds require special processing for analysis. More than one compound within each group can be analyzed in the same sample, but compounds outside the group are incompatible and cannot be analyzed within the same sample:

| | |
|---|---|
| Group I- | Ethyl Alcohol, Isopropyl Alcohol, and t-Butyl Alcohol |
| Group II- | n-Butyl Alcohol, s-Butyl Alcohol, iso-Butyl Alcohol and n-Propyl Alcohol |
| Group III- | Iso-Amyl Alcohol, Diacetone Alcohol, and Cyclohexanol |
| Group IV- | 2-Methoxyethanol, 2-Ethoxyethanol, and 2-Butoxyethanol |

**LAB SPECIFIC SAMPLES**

Each of the CIHLs has declared an area of expertise and it is recommended only these laboratories be used in those specialty areas. The areas and labs are:

| Area | Laboratory |
|---|---|
| Multi-element analysis using Inductive Coupled Plasma (ICP) Spectrometry. | Norfolk<br>San Diego |
| Advanced High Performance Liquid Chromatography. | San Diego |
| Gas Chromatography/Mass Spectrometry | Norfolk and San Diego |
| X-Ray Diffraction | Pearl Harbor |

## ROUTINE BIOLOGICAL SAMPLES

See page 12 of this Guide for general policies of sampling, packaging, labeling and shipping biologicals.

## BLOOD LEAD AND ZINC PROTOPORPHYRIN

Collect in one of the following Becton Dickinson (BD) Vacutainer Systems listed below:

| BD Number & Top Color | Description |
|---|---|
| 6488<br>Brown top | Sodium heparin tube for whole blood<br>(Specifically for blood lead determination) |
| 6527<br>Dark Blue top | Sodium heparin tube for whole blood<br>(Specifically for trace element studies) |
| 6541<br>Green top | Sodium heparin tube for plasma<br>(Specifically for plasma studies) |
| 6450<br>Lavender top | 15% EDTA tube for whole blood |

Samples must be thoroughly mixed with the heparin or EDTA immediately following collection. Keep samples refrigerated (do not freeze) and hand deliver or ship to the nearest laboratory using priority shipping methods. Use an insulated shipping container, such as a styrofoam shipper. For shipping long distances, freezer packs and express delivery are required.

## URINE MERCURY

See page 12 of this Guide for general policies of sampling, packaging, labeling and shipping biologicals.

By BUMED INSTRUCTION 6260.2, dated 7 November 1988, biological monitoring for mercury is no longer required. The potential for personnel exposure to elemental mercury vapor has been greatly reduced by the use of preencapsulated amalgams. Industrial hygiene surveys have shown routine use of preencapsulated amalgams does not result in overexposure of dental personnel to elemental mercury vapor. Therefore, by this BUMED instruction, neither biological sampling or air sampling is specifically required. Occasionally mercury urine may be prescribed by an occupational health professional as circumstances warrant.

Collect sample (first morning void, if possible) in the standard drug screening plastic bottle (NSN 6640-00-165-5778) and add 100 milligrams of potassium persulfate, a preservative. Please do not send more than 20 milliliters of urine per sample. Hand tighten the lid, and place each bottle in a zip-lok bag to contain any leakage during transit to the laboratory. Refrigerate during storage and ship, as soon as possible, in an insulated shipping container, using freezer packs (gel blocks) and express delivery.

## IMPINGER MEDIA PREPARATION

Impinger samples should be hand delivered to the laboratory. When this is not possible, the samples should be quantitatively transferred to 22 milliliter glass bottles with Teflon-lined caps (Supelco #2-3297M). Always provide the laboratory with a minimum of 20-30 milliliters of unexposed absorbing solution for a reagent blank.

The impinging solutions are:

| Analyte | Impinging Solutions |
|---|---|
| Ammonia | 0.01 N Sulfuric Acid (add 0.28 milliliters conc $H_2SO_4$ to 1 liter of double distilled or deionized water) |
| Cyanide | 0.1 N Potassium Hydroxide (add 5.6 grams KOH to 1 liter of double distilled water or deionized water) |
| Formaldehyde | 1% Sodium Bisulfite solution (1 gram $NaHSO_3$ in 100 milliliters of double distilled or deionized water) |
| Hydrazine | 0.1 N Hydrochloric Acid (add 8.6 milliliters of conc HCl to 1 liter of double distilled water or deionized water) |
| Sulfur Dioxide | 0.3 M Hydrogen Peroxide (add 17 ml of 30% $H_2O_2$ to one liter of double distilled or deionized water) |

NOTE: 20-30 ml of unexposed reagent is required for reagent blank for all impinging solutions above.

## SPECIAL SAMPLING & ANALYSES

### BULK SAMPLE SUBMISSIONS

The primary function of any industrial hygiene laboratory is the analysis of breathing zone air samples for contaminants. The CIHLs generally do not perform routine inventory environmental samples (e.g., heavy metals in paint, soil, water) or other bulk sample analysis to determine what components they contain or whether they meet manufacturer's specifications. Information for

the latter is available by writing the manufacturer and requesting product literature and Material Safety Data Sheets. Products for which this information is not available should not be used in the Navy system. Bulk samples should be submitted to the laboratories only under the following conditions:

(1) When the laboratory requests a bulk, as is required in the analytical method (e.g., PCBs, Naphthas, etc.).

(2) When all other means of obtaining information on the chemical composition of the material have been exhausted and prior approval has been given by the laboratory.

## CHROMIUM AND CHROMATES

Chromium metal, Cr (II) and Cr (III) compounds are collected on mixed cellulose ester filters (MCEF) and analyzed using atomic absorption spectrometry or ICP. Cr (VI) compounds cannot be determined if sampled on a MCEF.

Chromium in the +6 oxidation state (i.e., Cr (VI), chromic acid, chromium trioxide, all chromates and dichromates) must be collected on PVC filters, with backup pads. If other filter materials are used, the Cr (VI) may be reduced to the Cr (II) or Cr (III) states and thus give a reduced value for Cr (VI). Note: You no longer need to separate the filter from the backup pad prior to shipping the sample. Simply ship the PVC filters in their sampling cassettes.

## COAL TAR PITCH VOLATILES (CTPVs)/COKE OVEN EMISSIONS (COEs)/SELECTED POLYNUCLEAR AROMATIC HYDROCARBONS (PNAHs)

The coal tar pitch volatile test is designed for coal tar which has a high concentration of PNAHs. A summary of the test consists of:

1. Dissolving the sample in benzene and evaporating half the benzene mixture to determine how much benzene soluble is present. This part of the test is nonspecific, since almost all organics will dissolve in benzene.
2. To determine whether this material is a hazardous coal tar pitch or asphalt or just organic, a second test is performed on the remaining benzene-soluble fraction. It is analyzed for chrysene and benzo(a)pyrene using OSHA's test for PNAHs.

## SAMPLING CTPVs, COEs, AND SELECTED PNAHs

Air samples are collected by drawing known amounts of air through cassettes containing glass fiber filters (GFF). The recommended air volume is 960 liters at 2.0 LPM. Each GFF must be transferred to a separate vial after sampling and the vial sealed with a Teflon-lined cap. Samples must be protected from direct sunlight by wrapping aluminum foil around the vial.

## ANALYSIS OF CTPVs, COEs, AND SELECTED PNAHs

The filters are analyzed in the lab by extracting with benzene and gravimetrically determining the benzene-soluble fraction (BSF). If the BSF exceeds the appropriate exposure limit, the rest of the sample is analyzed by high performance liquid chromatography (HPLC) with a fluorescence or ultraviolet detector to determine the presence of selected PNAHs.

## INTERPRETATION OF (CTPV) STANDARD

OSHA interprets violation of the CTPV standard as exceeding 0.2 $mg/M^3$ of benzene-soluble material in air with the lab analysis of the benzene soluble fraction confirming the presence of benzo (a) pyrene and one or more of the five additionally named fused polycyclic hydrocarbons to which the standard refers: anthracene, acridine, pyrene, chrysene and phenanthrene. Sample for CTPV only when the work process involves or potentially involves the residues from heated and distilled tars derived from coal, petroleum, wood, shale oil or other organic materials. Asphalt is specifically exempted from the CTPV standard.

## ENVIRONMENTAL SAMPLES

All CIHLs are presently applying for AIHA accreditation in the National Lead Laboratory Accreditation Program (NLLAP) which was established by the U.S. Environmental Protection Agency (EPA) to evaluate and improve the performance of labs conducting analytical testing associated with lead abatement. When your CIHL is accredited it will then accept paint chips and dust wipes for lead analyses. Contact your local CIHL for a start-up date and sampling protocol. Since EPA methods emphasize outside environmental exposures over long periods of time, always contact your CIHL before sampling in a nontraditional, nonoccupational manner.

## FIBER COUNTS AND ASBESTOS IDENTIFICATION

These determinations are to be made in the field or at the local activity level. The CIHLs will assist on a case-by-case basis, however, prior approval for accepting these samples must be received from the CIHL before submitting fiber counts and asbestos identification samples to the CIHLs.

Laboratories performing asbestos tests must be proficient in the appropriate proficiency testing programs, (i.e., the Proficiency Analytical Testing (PAT) program for fiber counts and the NAVENVIRHLTHCEN's contractor-operated program for bulk asbestos identification).

## POLYCHLOROBIPHENYLS (PCBs)

All CIHLs routinely determine the PCB content in bulk samples as it relates to occupational health, with a lower reporting level of 0.1% or 1000 ppm. The laboratories do not routinely analyze to the EPA standard of 50 ppm for waste disposal purposes.

## SILICA (CRYSTALLINE SILICA) ANALYSIS

This method determines silica in respirable and total dust by the OSHA method. The sample filter used is a 5 um PVC filter. SKC Cat No. 225-8-01 (low silica homopolymer PVC), the Omega SILICAL PVC filters, or equivalent low silica homopolymer PVC filter should be used. The respirable dust sample is collected at 1.7 LPM for 800 to 1000 liter of air. A smaller air volume may be used if filter loading greater than 2.0 milligrams is expected.

Bulk samples can be semi-quantitatively analyzed for quartz and cristobalite.

## CORRECTION FACTORS

Certain metals (and cyanide) compounds in this guide are reported by the CIHLs "as" the metal ("as" the cyanide). The analytical instrument "sees" only the metal and ignores any other elements that make up the compound sampled. For example, if a sample of iron oxide ($Fe_2O_3$) is submitted for analysis, the lab reports the results "as Fe". If the analysis indicates a concentration of 10 micrograms iron per sample, this corresponds to a concentration of that amount of iron, not this amount of fume. It is the responsibility of the Industrial Hygienist who took/submitted the sample to convert the results to the correct weight of $Fe_2O_3$. This is done by dividing the molecular weight (MW) of the compound by the MW of the metal (or cyanide) contained. In this case the following apply:

MW of $Fe_2O_3$ divided by MW of $Fe_2$ = 159 divided by 111 = 1.4 (Correction Factor)

Multiplying the Correction Factor by the analytical results gives the correct weight of the contaminant.

1.4 times 10 micrograms iron = 14 micrograms of iron oxide

Correction Factors must be calculated and applied to all metal (and cyanide) compounds in this Guide which are reported "as ....".

## SOURCES OF ANALYTICAL SUPPLIES

NOTE: The mention of specific company names and products does not constitute endorsement by the Navy Environmental Health Center.

## MANUALS

The NIOSH analytical manuals may be obtained from:
http://www.cdc.gov/niosh/nmam/nmampub.html
Superintendent of Documents
PO Box 371954
Pittsburgh, PA 15250-7954

(202) 512-1800 or fax orders to (202) 512-2250.
[NIOSH Manual of Analytical Methods, fourth edition, 3 parts, Aug 94, DHHS Publication No. 94-113, price approximately $64.]

The OSHA analytical manuals may be obtained from:
http://www.osha-slc.gov/html/dbsearch.html
http://www.osha-slc.gov/dts/sltc/methods/index.html

ACGIH Publications
1330 Kemper Meadow Drive
Cincinnati, OH 45240-1634
Phone: (513) 742-2020 FAX: (513) 742-3355
[Publication #4542 and #4544 and #4545, Price approximately $445.]
http://www.acgih.org/

## FILTERS AND SORBENT TUBES

Filters and sorbent tubes may be obtained from a number of sources; however, this manual cites SKC order number for filters and tubes (listed in the SPECIAL INSTRUCTIONS column in the Laboratory Sampling Guide), simply because of convenience and uniformity.

Special attention should be given to SKC Guide to NIOSH/OSHA Air Sampling Standards which is in the SKC Comprehensive Catalog and Air Sampling Guide (Request free copy from SKC.)

SKC, Inc. World Headquarters
863 Valley View Road
Eight Four, PA 15330-9614
Phone: (800) 752-8472 FAX: (800) 752-8476
http://www.skcinc.com

SKC, Gulf Coast
9827 Whithorn Drive
Houston, TX 77095-5027

Phone: (800) 225-1309 FAX: (800)752-4853

SKC, West
P.O. Box 4133
Fullerton, CA 92634-4133
Phone: (800) 752-9378 FAX: (800) 752-1127

Supelco, Inc.
Supelco Park
Bellefonte, PA 16823-0048
Phone: (800) 247-6628 FAX: (800) 447-3044
Technical information only phone: (800) 359-3041
http://www.sigma-aldrich.com

Forest Biomedical
3975 South Main Street
Suite B
Salt Lake City, UT 84107
Phone: (801) 269-1327 FAX: (801) 269-1254

## PASSIVE MONITORS

3 M Company
Occupational & Environmental Safety Division
3 M Center, Bldg 224-5S-04
St. Paul, MN 55144-1000
Phone: (800) 752-3623 (Federal System Group orders)
Technical information only phone: (800) 243-4630
http://www.3m.com/market/government/

Landauer, Inc.
2 Science Road
Glenwood, IL 60425-1586
Phone: (708) 755-7000 FAX: (708) 755-7016
http://www.landauerinc.com/about.html

## PRINTED SAMPLE NUMBER LABELS

Shamrock Scientific
34 Davis Drive
Bellwood, IL 60104
Phone: (800) 323-0249 FAX: (800) 248-1907
http://www.shamrocklabels.com/